

Chemistry Practical Work

Part II : Examples of Expected Outcomes



Burning magnesium

Part II : Expected Outcomes

List of Experimental Work

1. Titrimetric Analysis: The Determination of a Strong acid with Standard Sodium Hydroxide.
2. Titrimetric Analysis: Standardisation of Hydrochloric acid using Sodium Carbonate.
3. Titrimetric Analysis: Estimating the Relative Molecular Mass of an Unknown Acid.
4. Gravimetric Analysis: To Determine the % Water of Crystallisation in a Hydrated salt.
5. The Preparation and Properties of Chlorine.
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21. Investigating Transition Metals.

The first twelve experiments are considered suitable for a first year A-level chemistry course.

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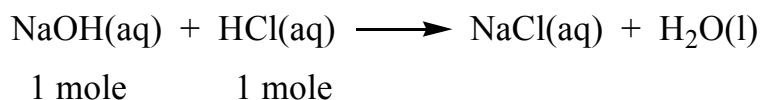
Practical One:

Titrimetric Analysis: The Determination of a strong acid with sodium hydroxide.

Example of Experimental Results:

Volume of HCl(aq) pipetted out: 25.00 cm ³	Burette readings		Molarity of sodium hydroxide solution used: 0.105M
Titration	Start cm ³	Final cm ³	Volume of sodium hydroxide solution delivered (final - start) cm ³
1	0.00	24.05	24.05
2	1.00	24.95	23.95
3	0.05	23.95	23.90
	Mean titration value:		23.97

Calculation:



Using formula:

$$\frac{M_1 \cdot V_1}{F_1} = \frac{M_2 \cdot V_2}{F_2}$$

$$\begin{aligned}M_1 &= 0.105 \text{ mol per dm}^3 \\V_1 &= 23.97 \text{ cm}^3 \\F_1 &= 1 \\M_2 &= \text{molarity of HCl} \\V_2 &= 25.00 \text{ cm}^3 \\F_2 &= 1\end{aligned}$$

$$\begin{aligned}0.105 \cdot 23.97 &= M_2 \cdot 25.00 \\M_2 &= \frac{0.105 \cdot 23.97}{25.00} = 0.101 \text{ mol per dm}^3\end{aligned}$$

From first principles:

$$\text{mol of NaOH used: } \frac{23.97}{1000} \times 0.105 \text{ mol} = 2.517 \times 10^{-3} \text{ mol}$$

$$\text{mol of HCl reacted: } 2.517 \times 10^{-3} \text{ mol}$$

(because the acid reacts with the alkali in a 1:1 ratio)

$$\begin{aligned}\text{This is contained in } 25.00 \text{ cm}^3 \text{ therefore, mol per dm}^3 &= \frac{1000}{25} \times 2.517 \times 10^{-3} \\&= 0.101 \text{ mol per dm}^3\end{aligned}$$

$$\text{(or, } 36.5 \times 0.101 = 3.687 \text{ g per dm}^3\text{)}$$

Practical Two: Standardisation of Hydrochloric acid using Sodium carbonate.**Example of Experimental Results:**

Mass of anhydrous sodium carbonate weighed out = 1.25 g

Volume of carbonate solution pipetted out: 25.00 cm ³	Burette readings		Molarity of sodium carbonate solution used: 0.0471M
Titration	Start cm ³	Final cm ³	Volume of hydrochloric acid solution delivered (final - start) cm ³
1	0.00	23.85	23.85
2	1.00	25.05	24.05
3	0.05	23.90	23.85
	Mean titration value:		23.92

Using formula: $\text{Na}_2\text{CO}_3(\text{aq}) + 2\text{HCl}(\text{aq}) \longrightarrow 2\text{NaCl}(\text{aq}) + \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g})$
 1 mole 2 mole

molarity of the carbonate solution: $\frac{\text{g per dm}^3}{\text{rel. mol. mass of carbonate}}$

$$\frac{5.00 \text{ g per dm}^3}{106 \text{ g per mol}} = 0.0471 \text{ mol per dm}^3$$

$$\frac{M_1 V_1}{F_1} = \frac{M_2 V_2}{F_2}$$

carbonate solution

$$\frac{0.0471 \times 25.00}{1} = \frac{M_2 \times 23.92}{2}$$

$$M_2 = \frac{2 \times 25.00 \times 0.0471}{23.92} = 0.0985 \text{ mol per dm}^3$$

From first principles: moles of sodium carbonate used in the titration =

$$\frac{25.00}{1000} \times 0.0471 = 1.178 \times 10^{-3} \text{ mol}$$

moles of hydrochloric acid reacting with this amount of carbonate =

$$2 \times 1.178 \times 10^{-3} \text{ mol} = 2.355 \times 10^{-3} \text{ mol}$$

This is contained in 23.92 cm³ of acid solution.

Therefore moles of acid per dm³ =

$$\frac{1000}{23.92} \times 2.355 \times 10^{-3} \text{ mol} = 0.0985 \text{ mol dm}^{-3}$$

Practical Three:

Estimate the relative molecular mass of an unknown acid.

Example of Experimental Results:

Mass of HX weighed out and dissolved in 250 cm³ of distilled water = 5.00g

Volume of HX solution pipetted out: 25.00 cm ³	Burette readings		Molarity of sodium hydroxide solution used: 0.099M
Titration	Start cm ³	Final cm ³	Volume of hydrochloric acid solution delivered (final - start) cm ³
1	0.00	24.85	24.85
2	0.00	24.05	24.05
3	0.00	24.70	24.70
	Mean titration value:		24.53

$$\text{molarity of HX solution} = \frac{5.00 \times 4}{\text{rmm of HX}} \text{ mol per dm}^3$$

$$\begin{aligned} \text{moles of sodium hydroxide used in titration} &= \frac{0.099 \times 24.53}{1000} \text{ mol} \\ &= 2.428 \times 10^{-3} \text{ mol} \end{aligned}$$

$$\text{moles of HX reacted with this amount of sodium hydroxide} = 2.428 \times 10^{-3} \text{ mol}$$

This is contained in 25.00 cm³ of HX solution therefore moles per dm³ =

$$2.428 \times 10^{-3} \times \frac{1000}{25} = 0.097 \text{ mol}$$

$$\text{Then, } \frac{5.00 \times 4}{\text{rmm of HX}} = 0.097$$

$$\& \text{ rel. molecular mass of HX} = 20/0.097 = 206.19$$

Practical Four:

To Determine the % water of crystallisation of a hydrated salt.

Example of Experimental Results:

	Mass (g)
Crucible + lid	23.65
Crucible + lid + copper(II) sulphate crystals	28.70
Copper(II) sulphate crystals used	5.05
Crucible + lid + anhydrous copper(II) sulphate	26.88
Crucible + lid + anhydrous copper(II) sulphate	26.87
Crucible + lid + anhydrous copper(II) sulphate	26.87
Anhydrous copper(II) sulphate	$26.87 - 23.65 = 3.22$
Water of crystallisation	$5.05 - 3.22 = 1.83$
% water of crystallisation	$(1.83/5.05) \times 100 = 36.23\%$

Practical Five: Preparation & Properties of Chlorine.

Example of Experimental Results:

Tests & Reactions	Observations	Inferences / Formulae / Equations
Potassium permanganate & concentrated hydrochloric acid	Vigorous reaction, at room temperature, producing green fumes & reddish brown solution	Chlorine formed. $2\text{KMnO}_4(\text{s}) + 16\text{HCl}(\text{aq}) = 2\text{KCl}(\text{aq}) + 8\text{H}_2\text{O}(\text{l}) + 2\text{MnCl}_2(\text{aq}) + 5\text{Cl}_2(\text{g})$
colour	Pale green	Colour is useful for identification purposes
smell	Pungent, choking poisonous	Smell useful for identification purposes The gas was used extensively in the 1st world war as a weapon
density	About twice as dense as air	Forms a layer below air. Flows along at ground level.
Solubility in water	Slightly soluble	Dissolves in water to give <i>chlorine water</i> . The solution contains, water, chlorine, hydrochloric acid and hypochlorous acid (chloric(I) acid).
Test:damp litmus paper	Blue litmus first turns red and then whitish (ie, bleached).	The chlorine decolourises the litmus dye in the litmus paper. $\text{Cl}_2(\text{g}) + \text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{HCl}(\text{aq}) + \text{HOCl}(\text{aq})$
Test: damp starch-iodide paper	The paper turns dark blue	The chlorine displaces iodine which combines with the starch to give a blue complex.
Test: shake the gas with potassium iodide or potassium bromide solutions.	In the case of potassium iodide solution the chlorine displaces iodine which colours the solution brown. With potassium bromide solution bromine is displaced and the solution is reddish brown.	eg, $2\text{KI}(\text{aq}) + \text{Cl}_2(\text{g}) \rightarrow 2\text{KCl}(\text{aq}) + \text{I}_2(\text{aq})$
Action of hydrocarbons	A reaction occurs producing dense black fumes. The fumes react with ammonia giving a dense fog.	Chlorine reacts with hydrocarbons when heated producing hydrogen chloride and carbon. The hydrogen chloride reacts with ammonia forming ammonium chloride.
Action of metals	Metals react vigorously with chlorine forming chlorides. Iron reacts producing sparks and dense brown fumes.	In the case of iron the brown fumes are due to fine particles of iron(III) chloride
Action of alkali. Chlorine bubbled into sodium hydroxide solution.	The chlorine reacts and a colourless solution is formed.	A bleach solution is produced. $\text{Cl}_2(\text{aq}) + 2\text{NaOH}(\text{aq}) \rightarrow \text{NaCl}(\text{aq}) + \text{NaOCl}(\text{aq}) + \text{H}_2\text{O}(\text{l})$

Practical Six:**Qualitative Analysis: Flame Test.****Example of Experimental Results:**

Test	Observations	Inferences
Flame test: substance A	A brilliant yellow orange colour was observed	Substance A is a sodium salt since the flame colour is indicative of sodium.
Flame test: substance B	A green colour was observed. The colour was dark green rather than pale green.	Substance B is a copper salt.
Flame test: substance C	A pale lilac colour was observed.	This suggests that substance C is a potassium salt.
Flame test: substance D	A brick red flame colour was observed.	Substance D is a compound containing calcium.
Flame test: substance E	A crimson red colour was observed. The colour was green through cobalt glass.	Substance E was a strontium salt.

Practical Seven A:
Tests for Gases & Vapours.

Example of Experimental Results.

Test	Observations	Inferences
Heat substance A in a borosilicate glass test tube over a bunsen flame.	Dense brown fumes were observed. When some of these fumes were taken up into a teat pipette and expelled into distilled water, the solution was acidic to litmus.	The brown gas is nitrogen(IV) oxide. Substance A could be a nitrate of a heavy metal (eg, lead, copper).
Substance B was added to dilute hydrochloric acid.	Vigorous evolution of a colourless gas. The gas turned lime-water milky	The gas was carbon dioxide and substance B is a carbonate or bicarbonate.
Substance C was treated with dilute hydrochloric acid.	A colourless gas was evolved which had a very pungent smell. The gas turned damp blue litmus red and turned potassium dichromate solution acidified with dilute sulphuric acid green.	The gas was sulphur dioxide. Substance C was a sulphite.
A white crystalline solid D was warmed with dilute sodium hydroxide solution.	A pungent, colourless gas was evolved. The smell was the same as that issuing from the dilute ammonia bottle. The gas formed dense white fumes with hydrogen chloride (from the concentrated hydrochloric acid reagent bottle). The gas turned damp red litmus blue.	The gas was ammonia. Substance D was an ammonium salt such as ammonium chloride.
Substance E was heated strongly in a borosilicate test tube.	The white crystalline solid melted and on continued heating decomposed producing a colourless gas. On cooling the contents of the tube crystallised to form a pale yellow solid. The gas was odourless and relit a glowing splint.	The gas was oxygen. Substance E was probably an alkali metal nitrate (eg, sodium nitrate in which case the residue was sodium nitrite).

Practical Seven B:

Tests for Cations in aqueous solution.

Example of Experimental Results:

Test	Observations	Inferences
(i) Substance A was dissolved in water, in a test tube, to give a dilute solution. A few drops of 10% ammonium carbonate solution was added. (ii) Dilute sodium hydroxide solution was added to a further sample of a solution of A. (iii) The solution of A was tested with litmus paper.	The solution was colourless. A white precipitate was formed. A small amount of white precipitate was formed which was insoluble in excess hydroxide solution. The solution was neutral to litmus.	Substance A is a soluble calcium salt.
(i) In a test tube a few crystals of substance B was dissolved in distilled water. Dilute sodium hydroxide was added. (ii) ammonia solution was added to a dilute solution of substance B until the ammonia solution was in excess.	Crystals of substance B were blue. They dissolved giving a blue solution. A pale blue precipitate was formed when sodium hydroxide solution was added to a solution of B. Adding ammonia solution resulted in a pale blue precipitate which dissolved in excess to give a clear, dark blue solution.	Many copper salts are blue (eg, copper(II) sulphate). Copper(II) hydroxide is formed. Copper(II) hydroxide is produced initially. This dissolves forming the soluble, dark blue, tetraamminecopper(II) ion.
Colourless crystals of C were dissolved in water to give a dilute solution. (i) to a few cm ³ of this solution was added a few drops of dilute sodium carbonate solution. (ii) to a few cm ³ of a solution of C was added dilute sulphuric acid.	 A white precipitate formed. A white precipitate formed.	 The cation of substance C forms, white, insoluble carbonate and sulphate. Substance C could be a soluble barium salt (eg, barium chloride).
Substance D consists of a pale green crystalline solid. (i) in a test tube, make a dilute solution in distilled water. Add dilute sodium hydroxide solution to 2 cm ³ of D. (ii) in a borosilicate glass test tube heat D, at first gently and then strongly.	The colour is reminiscent of iron(II) salts. The salt is freely soluble in water. Adding alkali produces a green precipitate which slowly turns yellow-brown. Substance D decomposes producing a colourless liquid on the cool part of the tube. Heating to a higher temperature causes further decomposition leaving a red-brown solid and producing a colourless, acidic, gas (turns damp blue litmus red)	Substance D is possibly hydrated iron(II) sulphate. This is a pale green solid and decomposes giving water vapour and then sulphur oxides and leaving iron(III) oxide. Iron(II) sulphate is soluble in water and the solution gives insoluble iron(II) hydroxide (insoluble in excess alkali). This hydroxide is air oxidised to brownish iron(III) hydroxide.

Practical Seven C:

Tests for Anions in aqueous solution.

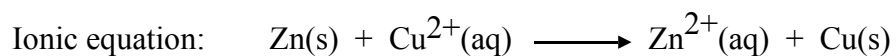
Example of Experimental Results:

TEST	OBSERVATION	INFERENCE
A dilute solution of substance A was added to a test tube. 1 cm ³ of dilute silver nitrate was added followed by 2 cm ³ of dilute nitric acid.	A cream coloured precipitate was formed. Excess dilute ammonia solution was added and the mixture shaken. The cream precipitate dissolved to give a clear solution.	The precipitate is probably silver bromide. Silver bromide is confirmed. Substance A is a soluble bromide.
A dilute solution of substance B was added to a test tube. Dilute barium chloride solution was added. The mixture was made acid with dilute hydrochloric acid.	A white precipitate was formed. Following addition of hydrochloric acid, the white precipitate dissolved with evolution of a colourless, pungent smelling, gas. The gas turns acidified dichromate solution green.	The white precipitate was barium sulphate <u>or</u> sulphite. Sulphite is indicated because sulphites are decomposed by dilute acid giving sulphur dioxide. This gas turns dichromate solution green.
A dilute solution of substance C was prepared in a test tube. The solution was made alkaline with dilute sodium hydroxide solution. A small amount of aluminium powder was added and the mixture warmed.	A small volume of gas was evolved (as evidenced by the formation of bubbles in the aqueous mixture). The gas was colourless but had a pungent smell. The gas turned damp red litmus blue. The gas also produced white fumes with hydrogen chloride (from the mouth of the concentrated hydrochloric acid bottle).	The gas was ammonia. This indicates that substance C contains nitrogen, probably in the form of nitrate ion.
A small quantity of substance D was introduced into a test tube. Dilute hydrochloric acid was added, a little at a time. Substance D was insoluble in distilled water.	Vigorous evolution of a colourless gas was evolved. This gas was odourless and did not change the colour of litmus paper. Passing the gas into lime water produced a milky solution.	The gas was carbon dioxide. Substance D was an insoluble carbonate (such as calcium carbonate).

Practical Eight:

Measurement of the Enthalpy change when a metal is displaced from solution.

Example of Experimental Results:



First set of data:

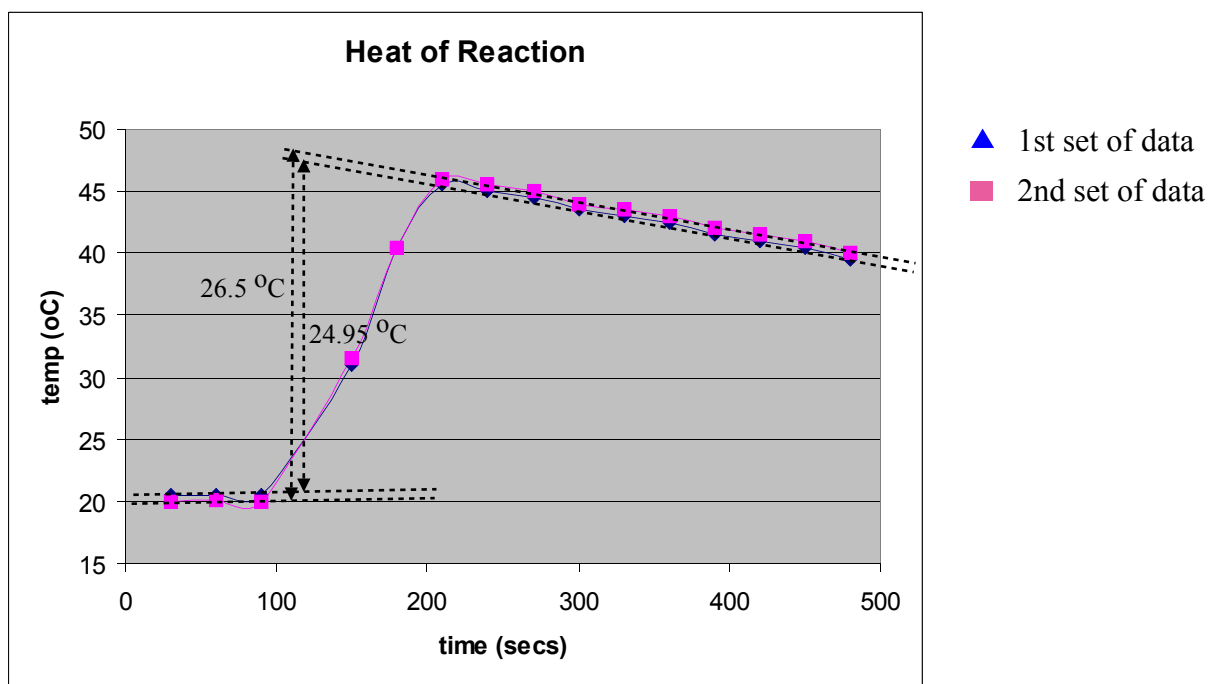
Time (secs)	Temperature (°C)
30	20.5
60	20.5
90	20.5
150	31.0
180	40.5
210	45.5
240	45
270	44.5
300	43.5
330	43
360	42.5
390	41.5
420	41.0
450	40.5
480	39.5

← zinc powder added

Second set of data:

Time (secs)	Temperature (°C)
30	20.0
60	20.1
90	20.0
150	31.5
180	40.5
210	46.0
240	45.5
270	45.0
300	44.0
330	43.5
360	43.0
390	42.0
420	41.5
450	41.0
480	40.0

← zinc powder added



$$A_1 \text{ (heat change from the 1st set of data)} = 26.5 \times 50 \times 4.2 = 5565 \text{ J}$$

$$A_2 \text{ (heat change from the 2nd set of data)} = 28.0 \times 50 \times 4.2 = 5880 \text{ J}$$

Assuming that the original copper(II) sulphate was 0.5 M, heat changes per mole:

$$1 = 5565 \times 40 = 223 \text{ kJ mol}^{-1}$$

$$2 = 5880 \times 40 = 235 \text{ kJ mol}^{-1}$$

$$\text{Mean heat change} = 229 \text{ kJ mol}^{-1}$$

$$\text{ie, } \Delta H = 229 \text{ kJ mol}^{-1}$$

Answers to Questions:

1. To ensure that the zinc is at the same temperature as the copper sulphate solution at the start.
2. To establish the temperature before the reaction starts.
3. The corrected temperature rise is an estimate of the temperature rise assuming no heat transfer from the calorimeter.
4. Heat loss from the calorimeter to the surroundings during the course of the experiment.
This may not be totally compensated for by the corrected temperature rise value.
Human error in making up copper(II) solution and taking temperature readings, etc.
Less than 100% reaction between zinc and copper(II) ions resulting in a low temperature rise value.

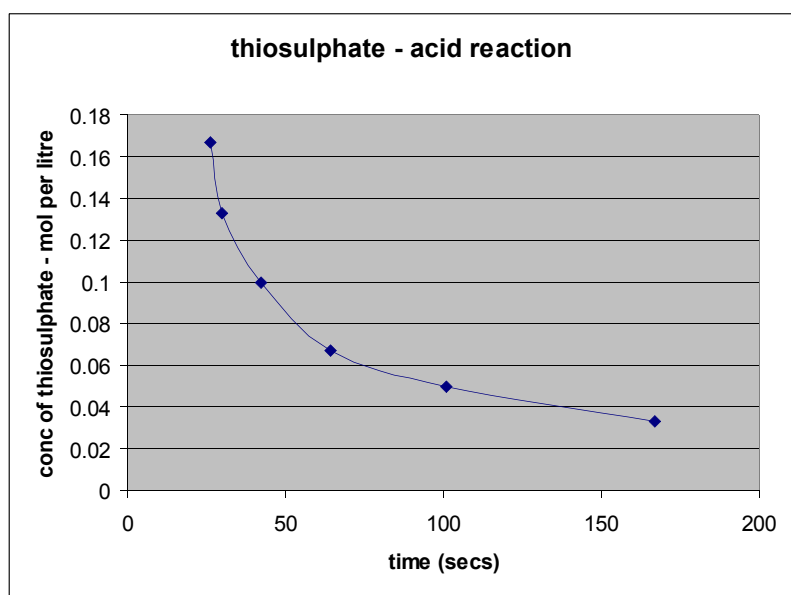
Practical Nine:

Chemical Kinetics Investigations: Examples of Experimental Results:

(A) To investigate the effect of concentration on the speed of reaction and to find the rate equation for a reaction.

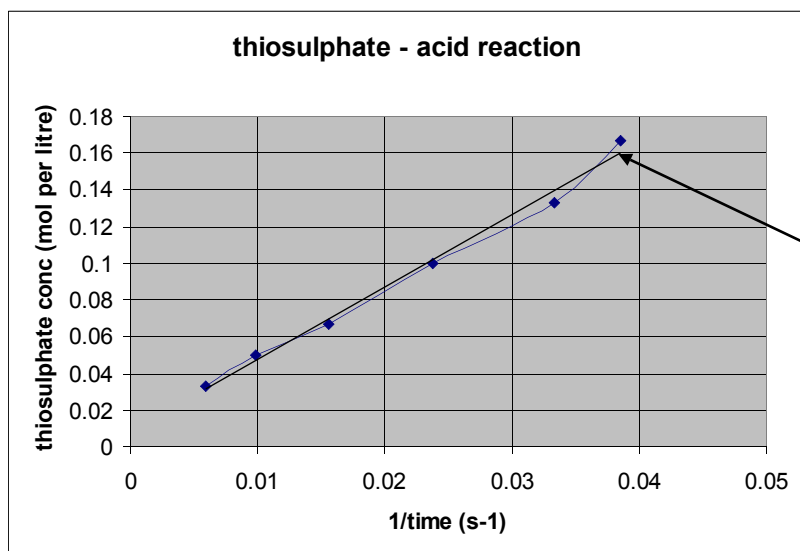
Effect of concentration of thiosulphate on reaction rate:

time (secs)	thiosulphate conc (mol per litre)
26	0.167
30	0.133
42	0.1
64	0.067
101	0.05
167	0.033



Shows that as the concentration of the thiosulphate decreases the reaction takes longer.

reciprocal time (s-1)	thiosulphate conc (mol per litre)
0.0385	0.167
0.0333	0.133
0.0238	0.1
0.0156	0.067
0.0099	0.05
0.00599	0.033



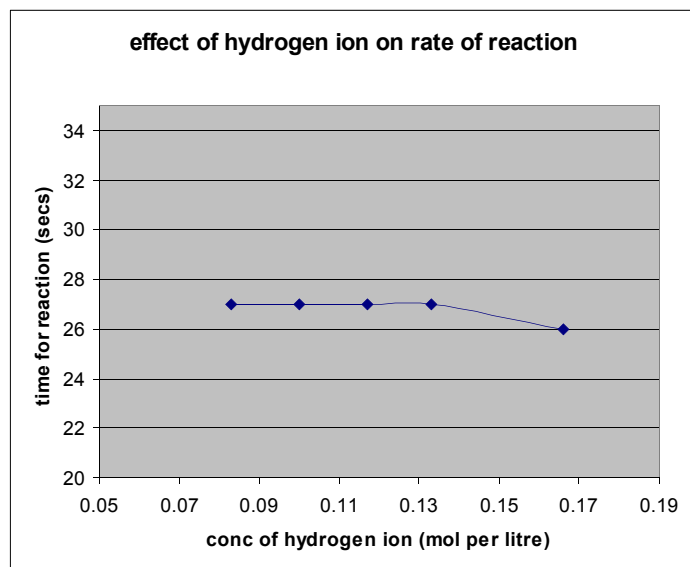
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Shows that the rate of reaction is directly proportional to the thiosulphate concentration

$$\text{rate} = k \cdot \text{concentration}$$

Effect of concentration of hydrogen ion on rate of reaction:

conc of hydrogen ion (mol per litre)	time of reaction (secs)
0.166	26
0.133	27
0.117	27
0.1	27
0.083	27



These results show that changing the concentration of the acid has little or no effect on rate, ie, non rate determining.

Therefore overall,

$$\text{rate} = k [\text{thiosulphate}]^1 [\text{hydrogen ion}]^0$$

$$\text{rate} = k [\text{thiosulphate}]$$

Value of rate constant (k):

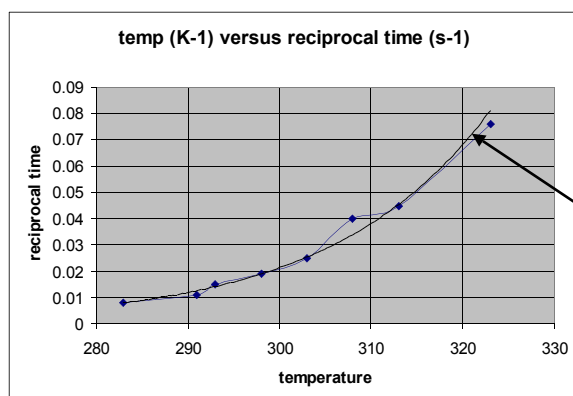
(Using data from the second graph on the previous page)

$$\begin{aligned} k &= \text{rate}/\text{concentration} = \frac{0.04 \text{ mol dm}^{-3} \text{ s}^{-1}}{0.16 \text{ mol dm}^{-3}} \\ &= 0.25 \text{ s}^{-1} \end{aligned}$$

(B) Investigating the effect of Temperature on the Rate of Chemical Reaction.

Example of Experimental Results:

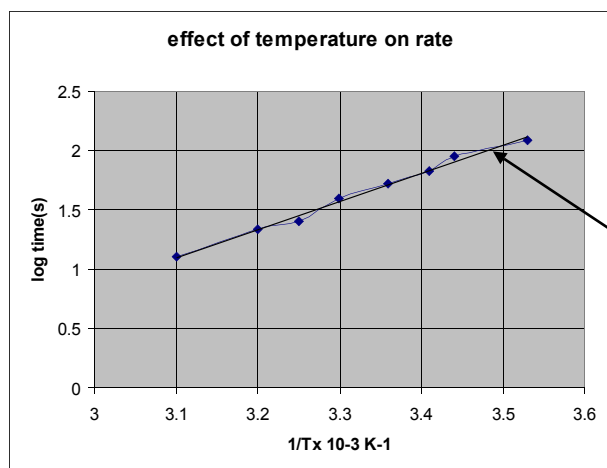
temp °K	1/time s ⁻¹
283	0.0082
291	0.011
293	0.015
298	0.019
303	0.025
308	0.04
313	0.045
323	0.076



best line through points

This shows a steady increase in rate with increase in temp. However, rate is **not** directly proportional to temperature. The relationship is more complex.

time (s)	Temp K ⁻¹	1/T 10 ⁻³	log t
122	283	3.53	2.09
90	291	3.44	1.95
67	293	3.41	1.83
52	298	3.36	1.72
40	303	3.3	1.6
25	308	3.25	1.4
22	313	3.2	1.34
13	323	3.1	1.11



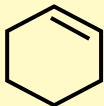
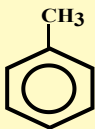
best straight line through the points

$$\text{Slope} = 2.31 \times 10^3 = E / 2.303 \times R$$

$$\text{Activation energy (E)} = 2.31 \times 10^3 \times 2.303 \times 8.31 = 44.21 \text{ KJ mol}^{-1}$$

Practical Ten:
Reactions of Hydrocarbons

Example of Experimental Results:

Hydrocarbon	Combustion	Permanganate (dilute aqueous solution)	Halogen (bromine water)	Conc. Sulphuric acid	Comments
Hexane $(\text{CH}_3(\text{CH}_2)_4\text{CH}_3)$	When a few drops are ignited on a crucible lid (in a fume cupboard) the liquid hydrocarbon burns readily with a clean flame	When a few drops of the hydrocarbon are added to 2 cm ³ of very dilute potassium permanganate & the mixture shaken, the hydrocarbon floats to the surface of the aqueous solution & the latter remains pink indicating no reaction.	When shaken with dilute bromine water there is no apparent reaction. The hydrocarbon floats to the surface of the bromine water forming a colourless layer. In the presence of bright sunlight the hydrocarbon reacts slowly decolourising the bromine water.	When carefully shaken with concentrated sulphuric acid the hydrocarbon does not react. After a time it separates from the acid solution unchanged.	Highly flammable but markedly unreactive towards the test reagents.
Cyclohexene 	This colourless, rather unpleasant smelling liquid, burns readily on a crucible lid producing a luminous smokey flame.	Shaking a few drops of this hydrocarbon with a couple of cm ³ of dilute potassium permanganate solution results in immediate reaction and removal of the pink colour due to permanganate ion.	The hydrocarbon reacts immediately with the bromine water dispelling the characteristic red-brown colour of bromine leaving a colourless mixture.	The hydrocarbon reacts vigorously with concentrated sulphuric acid forming soluble hydrogen sulphate and brown resinous material. The mixture gets warm.	Cyclohexene is a simple alkene. Alkenes are much more reactive than alkanes. With permanganate they form diols, with bromine they form 1,2-dibromides & with sulphuric acid they form a mixture of hydrogen sulphates and resinous material.
Toluene (methylbenzene) 	This aromatic hydrocarbon burns with a very luminous, smokey, flame.	There is no reaction with dilute permanganate.	There is no reaction with bromine water.	Little reactivity towards concentrated sulphuric acid at room temperature.	Aromatic hydrocarbons are generally less reactive than alkenes but more reactive than alkanes. At elevated temperatures toluene is oxidised by, concentrated permanganate solution, to benzoic acid. It is substituted by bromine and sulphonated by concentrated sul-

By applying these tests it is possible to distinguish hydrocarbons belonging to the alkane, alkene and arene series. Alkanes burn with relatively clean flames where as alkenes and arenes burn with luminous smokey flames. To distinguish between alkenes and arenes use dilute permanganate solution, dilute bromine solution and concentrated sulphuric acid at room temperature (see above). Arenes usually have a fairly pleasant odour described as aromatic or woody; alkenes are more volatile and have less pleasant odours.

Practical Eleven:

Properties and reaction of Alcohols.

Example of Experimental Results:

Test	Observation	Conclusion
Solubility in water	The simple alcohols, such as methanol and ethanol, are freely soluble in water. The solutions have a neutral pH (ie, not acidic or basic).	The alcohols have polar molecules which have a strong affinity with polar water molecules (largely through hydrogen bonding). The higher alcohols which have larger hydrocarbon components to their molecules have less affinity with water and are less soluble.
Oxidation with acidified potassium dichromate solution.	After warming in hot water for a few minutes the dichromate-alcohol reaction mixture changes colour from orange to green. The vapours issuing from the test tube have a pungent ripe apple odour.	Methanol is not oxidised under these conditions. Ethanol is readily oxidised first to ethanal (which has the detected odour) and then to ethanoic acid. The latter gives the volatile reaction vapours acidic properties (ie, turns damp blue litmus red). A secondary alcohol, such as butan-2-ol under these conditions, would be oxidised to a ketone; butan-2-ol gives propanone.
A small quantity of phosphorus pentachloride was added to a couple of cm ³ of alcohol in a test tube.	A violent reaction ensued. A misty vapour was produced which formed a white smoke with ammonia (from the ammonia treated filter paper). The reaction mixture remained colourless but did get warm.	Alcohols react with PCl ₅ forming chloroalkanes. Ethanol for example gives chloroethane. Hydrogen chloride and phosphorus oxychloride are the other products. The hydrogen chloride vapour reacts with ammonia to give ammonium chloride (which gives rise to the white smoke).
A small piece of sodium metal was added to a couple of cm ³ of methanol.	The metal reacted steadily with the alcohol producing bubbles of gas. A clear colourless solution remained.	The gas is hydrogen. The remaining solution contains sodium methoxide. This reaction is useful as a simple test and also for disposing of small amounts of waste sodium metal. Adding the waste sodium directly to water causes a more vigorous reaction which can be violent or even explosive causing molten sodium to spit out of the reaction vessel. $2\text{Na(s)} + 2\text{CH}_3\text{OH(l)} = 2\text{CH}_3\text{ONa(alc)} + \text{H}_2\text{(g)}$

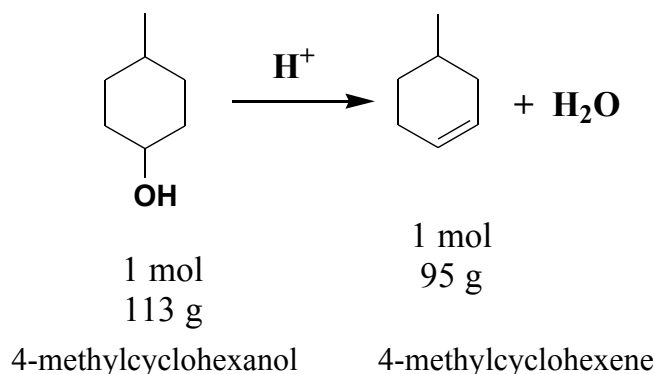
Practical Twelve:

Preparation of an Alkene by dehydration of an Alcohol.

Example of Experimental Results:

% Yield:

According to the balanced equation 1 mol of alcohol will give a maximum yield of 1 mol of alkene.



Let us assume that 10 g of purified alkene was obtained (ie, after separation and purification, by washing and distillation).

0.175 mol of alcohol was used so the maximum yield of alkene would be 0.175 mol or 16.6 g.

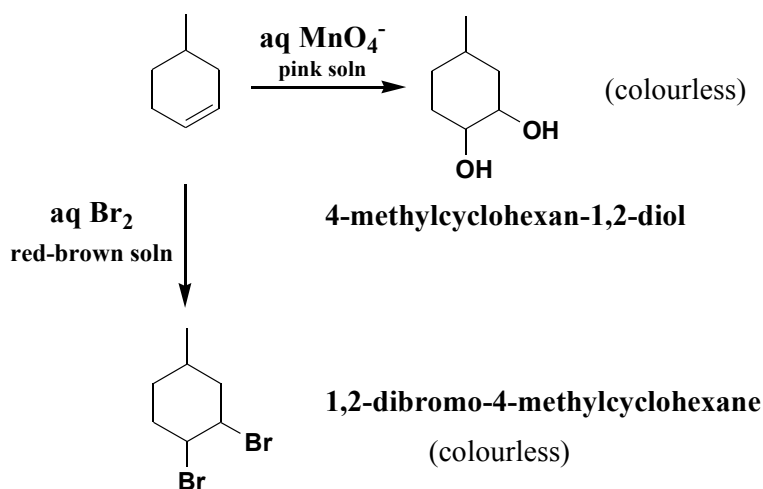
Therefore,

$$\% \text{ yield} = \frac{10}{16.6} \times 100 = 60.2 \%$$

The reason the yield is less than 100% is because, (i) incomplete reaction and material degradation (as evidenced by a certain amount of browning of the reaction mixture), (ii) loss of product in the work-up procedure (ie, washing and distillation); not all of the product can be retrieved from the wash solutions and distillation apparatus also some product evaporation occurs.

When tested with permanganate and bromine solutions the alkene reacted readily decolourising both of the test reagents.

ie,



Practical Thirteen:

Using Permanganate to find the amount of Water of Crystallisation in an Iron salt

Example Results:

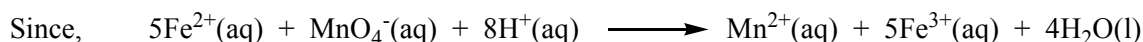
Mass of ammonium iron(II) sulphate per 250 cm³ of solution = 6.05 g

Volume of iron(II) sulphate solution pipetted out: 25.00 cm ³	Burette readings		Molarity of potassium permanganate solution used: 0.021M
Titration	Start cm ³	Final cm ³	Volume of potassium permanganate solution delivered (final - start) cm ³
1	0.00	14.85	14.85
2	0.05	14.75	14.70
3	0.05	14.80	14.75
	Mean titration value:		14.77

molarity of the ammonium iron(II) sulphate solution =

$$\frac{6.05 \times 4}{284 + x18} \quad (\text{where } x = \text{moles of water of crystallisation})$$

$$\begin{aligned} \text{moles of potassium permanganate used in the titration} &= \frac{14.77}{1000} \times 0.021 \\ &= 3.10 \times 10^{-4} \text{ mol} \end{aligned}$$



moles of iron(II) reacting with this amount of permanganate =

$$5 \times 3.10 \times 10^{-4} = 1.55 \times 10^{-3} \text{ mol}$$

$$\text{moles of iron(II) per dm}^3 \text{ of solution} = \frac{1000}{25} \times 1.55 \times 10^{-3} = 0.062 \text{ mol}$$

$$\text{Then, } \frac{6.05 \times 4}{284 + x18} = 0.062$$

from which $x = 5.9$

therefore, $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$

Practical Fourteen:

Preparation of Iodoform

Example of Experimental Results:

From the given equation 1 mol of propanone gives 1 mol of iodoform.
0.02 mol of propanone was used; 0.02 mol (7.88 g) of iodoform would be the maximum yield (ie, 100%).
However, 100% is unrealistic because even if the reaction was quantitative some product would be lost in the isolation and purification procedures.

Lets assume 0.5 g of pure, dry, product was obtained.

$$\text{Then \% yield} = \frac{0.5}{7.88} \times 100 = 6.3 \% \quad (\text{which is low!})$$

Iodoform is a yellow crystalline solid with an antiseptic odour. It melts at 119°C with some decomposition.

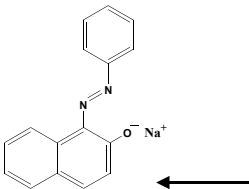
Note: The reagents used in this reaction are often used to test for CH₃CO- and CH₃CHOH- groups in samples submitted for analysis. If an unknown compound contains these functional groups the compound will react with iodide and hypochlorite to give a yellow precipitate of iodoform.

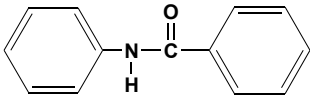
Practical Fifteen:

Properties & Reactions of Carbonyl Compounds, Carboxylic Acids & Amines.

Example of Experimental Results:

Test	Observation	Inference
CARBONYL COMPOUNDS		
Fehlings A & B were mixed and a small quantity of ethanal added. The mixture was warmed for a few minutes in hot water.	A red-brown precipitate was formed.	Ethanal is oxidised by the reagent to ethanoic acid. The precipitate is hydrated copper(I) oxide. This result is typical of aldehydes. Ke-
A drop of ethanal was added to <i>freshly prepared</i> Tollen's reagent.	An immediate grey-black precipitate was formed. When left to stand a silver mirror was observed on the inside wall of the test tube. No reaction with propanone.	This is sometimes referred to as the silver mirror test . It is sensitive for aldehydes. Ketones do not react. The aldehyde is oxidised to the corresponding carboxylic acid, ie, ethanoic acid in this case.
A few drops of ethanal were added to 2 cm ³ of Brady's reagent.	An immediate yellow-orange precipitate was formed. A similar precipitate was formed using propanone instead of ethanal. However, the reaction occurred more slowly.	The precipitates were the corresponding hydrazones . This is a good test for the carbonyl group. It does not help to distinguish aldehydes from ketones since they both contain the carbonyl group and react with the reagent.
CARBOXYLIC ACIDS		
solubility	Ethanoic acid is freely soluble in water. The solution is acidic to litmus and pH paper (ie, turns the papers red). Benzenecarboxylic acid (benzoic acid) is sparingly soluble in cold water. It is soluble in hot water. It is soluble in methylbenzene (toluene)	Carboxylic acids have polar molecules and are soluble in polar solvents to varying degrees. Benzenecarboxylic acid molecules have a large non-polar hydrocarbon group (ie, the benzene ring) which reduces their solubility in water. However, the benzene ring enhances the solubility of the acid in the aromatic solvent methylbenzene
Benzenecarboxylic acid (benzoic acid) was added to dilute sodium hydroxide.	The acid dissolves readily in the alkali to give a clear, colourless, solution. Adding dilute hydrochloric acid to excess produces a white precipitate.	The acid reacts with the alkali forming the soluble acid salt, sodium benzenecarboxylate (sodium benzoate). When strong mineral acid is added the carboxylate ion is protonated and the free benzoic acid precipitated (the hydrochloric acid is the stronger acid and displaces the weaker organic acid from solution).
Ethanoic acid was added to dilute sodium carbonate solution. The test was repeated with the aromatic acid, benzoic acid.	An immediate reaction occurred with rapid evolution of a colourless gas. A similar reaction occurred when benzoic acid was tested. However, in the latter case, the reaction occurred much more slowly.	A reaction occurred to give sodium ethanoate and carbon dioxide. In the case of benzoic acid, sodium benzoate was formed and carbon dioxide evolved slowly.
Ethanoic acid was heated with (a) pentanol and (b) ethanol. In both cases concentrated sulphuric acid was added as catalyst and dehydrating agent.	(a) when the reaction mixture was added to dilute carbonate vigorous evolution of gas occurred and an odour of bananas was observed. (b) vigorous evolution of gas was again observed and a fruity odour was observed (the odour was also reminiscent of nail varnish remover!).	(a) the odour was due to the ester pentyl ethanoate. The gas was carbon dioxide generated from the reaction between carbonate and sulphuric acid. The gas helps to lift the odoriferous vapours out of the reaction vessel towards the experimenter! (b) the odour was due to the ester, ethyl ethanoate. The gas was carbon dioxide.

TEST	OBSERVATION	INFERENCE
CARBOXYLIC ACIDS (continued)		
Reaction of acids with neutral iron(III) chloride solution.	(a) a dark reddish solution was produced. When heated a reddish precipitate was formed. (b) a buff coloured precipitate was formed. This turned white when dilute mineral acid was added.	(a) iron(III) ethanoate (acetate) was formed. (b) iron(III) benzenecarboxylate (benzoate) was formed initially, this was converted to insoluble benzenecarboxylic acid (benzoic acid) when treated with mineral acid.
Reaction of ethanoic acid (acetic acid) with phosphorus pentachloride.	Ethanoic acid reacted vigorously. Hydrogen chloride was evolved. This formed white fumes with ammonia and turned damp blue litmus paper red.	Ethanoic acid reacts forming ethanoyl chloride and hydrogen chloride.
AMINES		
Aqueous methanamine (methyl amine).	This solution has a fishy smell. The solution is alkaline (pH=9). (the vapour from this solution is flammable - burns with a pale blue flame)	Simple amines have a fishy and/or ammonia type odour. They are organic bases. The simple ones are soluble in water with which they form an equilibrium mixture in which some of the amine molecules are in protonated form (eg, CH_3NH_3^+)
(a) Test benzenamine (aniline) with dil. HCl. (b) Test benzenamine with dil. sodium hydroxide.	(a) Benzenamine dissolves in hydrochloric acid. (b) Benzenamine is insoluble in dil. sodium hydroxide.	Benzenamine is an aromatic primary amine. It is sparingly soluble in water but much more soluble in dilute mineral acid. This is because it reacts with the acid forming an amine salt. eg, $\text{C}_6\text{H}_5\text{NH}_2(\text{l}) + \text{aqHCl} = \text{aqC}_6\text{H}_5\text{NH}_3^+ + \text{aqCl}^-$
Ice cold methanamine was added to an ice cold solution of nitric (III) acid (nitrous acid). Cold benzenamine was added to ice cold nitric(III) acid solution. An alkaline solution of 2-naphthol was then added.	Vigorous evolution of a colourless gas occurred. The gas did not support combustion & did not turn lime water milky. A red precipitate was formed.  a red azo dye	A reaction occurs to give methanamine diazonium chloride. This is highly unstable and immediately decomposes giving methanol and nitrogen gas. (aliphatic amines do not form stable diazonium compounds and therefore do not give azo dyes) Aromatic amines like benzenamine react with nitric(III) acid solution, at temperatures below 5°C , forming relatively stable diazonium compounds. These react with phenols forming azo dyes. In this case the product is sodium 2-phenylazonaphthate

TEST	OBSERVATION	INFERENCE
Amides		
An alkaline solution of benzenamine (aniline) was shaken with 0.5 cm ³ of benzenecarbonyl chloride (benzoyl chloride)	A grey precipitate was formed. This was filtered off and recrystallised from IMS.	<p>The recrystallised product was a white crystalline solid which melted at 163°C.</p> <p>This corresponds to N-phenylbenzenecarboxamide (benzanilide).</p> <p>The pure dry product weighed; 0.40 g. This compares with 1.06 g for 100% yield therefore a yield of about 37% was achieved.</p>
<div style="text-align: center;">  <p>benzanilide</p> </div>		

Example of Experimental Results:

[illegible]

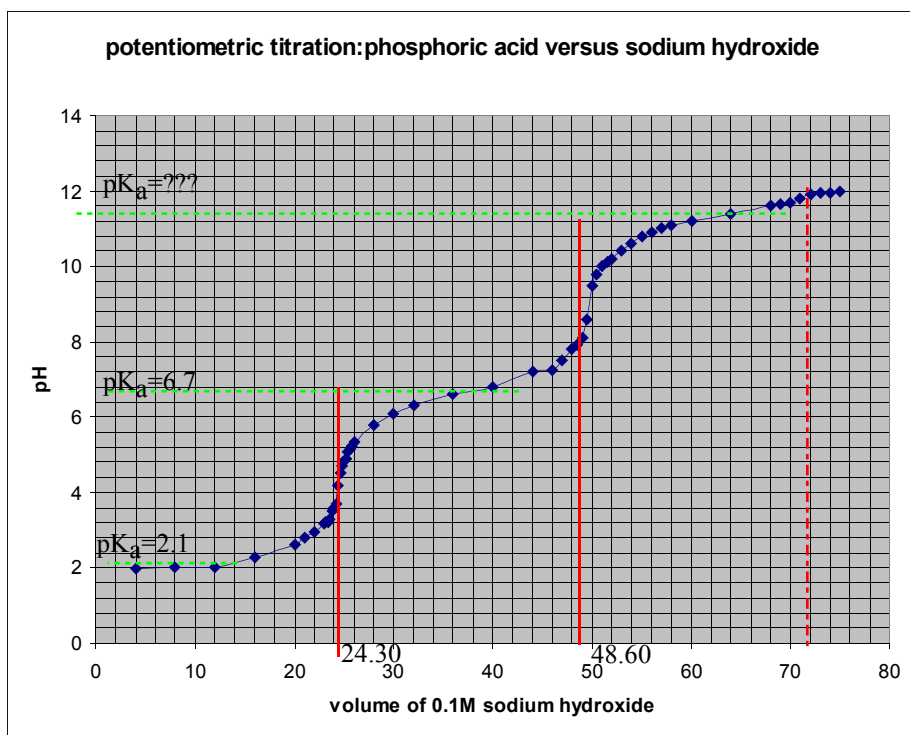
Practical Sixteen:

Potentiometric Titration:

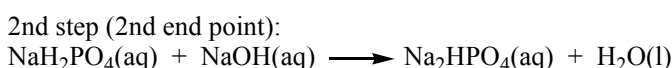
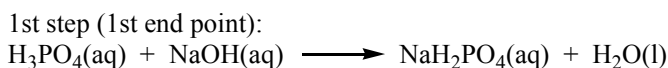
Example of Experimental Results:

Potentiometric titration of approximately 0.1M phosphoric acid against 0.10M sodium hydroxide. In this experiment 20.00 cm³ of the acid was titrated against the alkali.

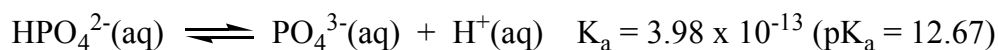
0.1M	pH		
		44	7.2
4	1.98	46	7.25
8	2	47	7.5
12	2	48	7.8
16	2.27	48.5	7.9
20	2.6	49	8.1
21	2.8	49.5	8.6
22	2.95	50	9.5
23	3.18	50.5	9.8
23.2	3.2	51	10
23.4	3.2	51.5	10.1
23.6	3.28	52	10.2
23.8	3.5	53	10.4
24	3.6	54	10.6
24.2	3.7	55	10.8
24.4	4.2	56	10.9
24.6	4.5	57	11
24.8	4.7	58	11.1
25	4.8	60	11.2
25.2	4.9	64	11.4
25.4	5.08	68	11.6
25.6	5.15	69	11.65
25.8	5.22	70	11.7
26	5.35	71	11.8
28	5.8	72	11.9
30	6.08	73	11.95
32	6.3	74	11.96
36	6.6	75	11.98
40	6.8		



The results indicate that the first end point is at 24.50 cm³. This gives a molarity of 0.123M for the phosphoric acid. Two end points are clearly visible corresponding to,

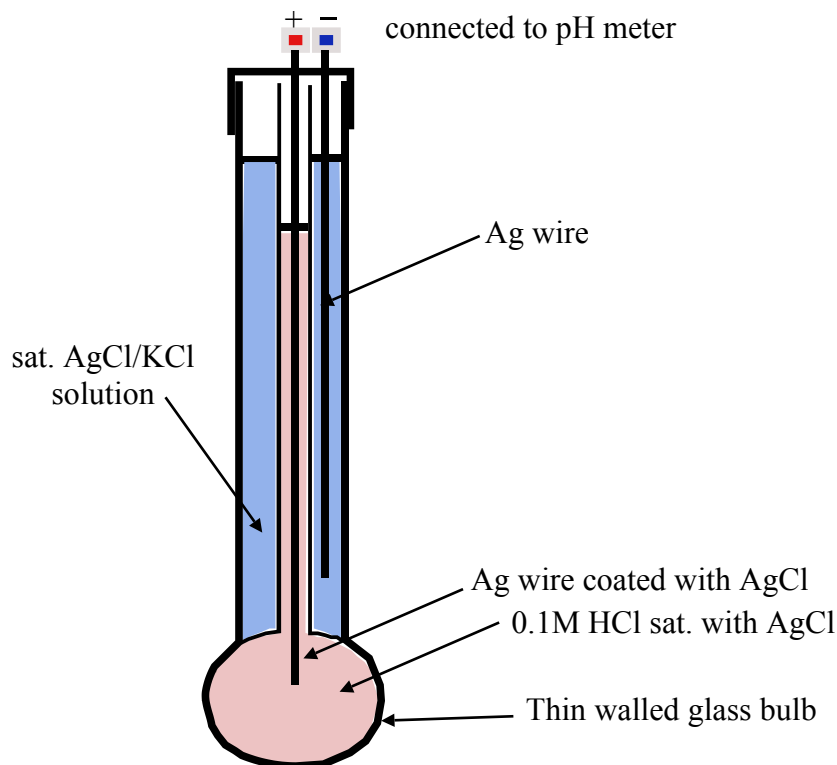


The third end point is not visible since the step (ie, change in pH) is very small and cannot be detected using this equipment (it should occur at pH 12 - 13 since the literature value for the pK_a is about 12.7).



Answers to Questions:

1. Combination glass/silver chloride electrode.
(ie, pH sensitive glass electrode + silver chloride reference electrode)

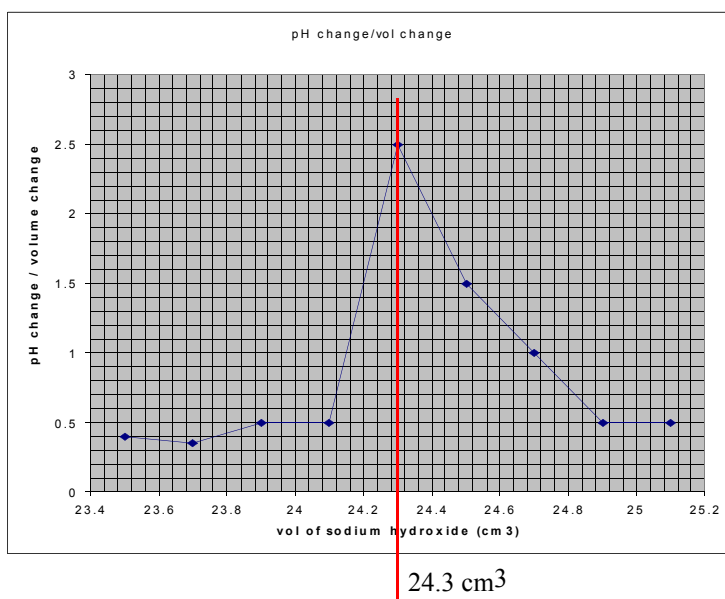


2. Not reliant on colour changes. This could be useful if you happen to be colour blind or unfamiliar with the common indicators and their characteristic colour changes! Also, the analytical sample may be coloured and this colour may obscure the indicator colours. A simple example of this would be the analysis of brown vinegar for ethanoic acid. Vinegar contains about 4 - 5% ethanoic acid and it can be estimated by potentiometric titration against dilute sodium hydroxide.

3. First derivative method.

Using the previous titration figures as an example.

First end point:

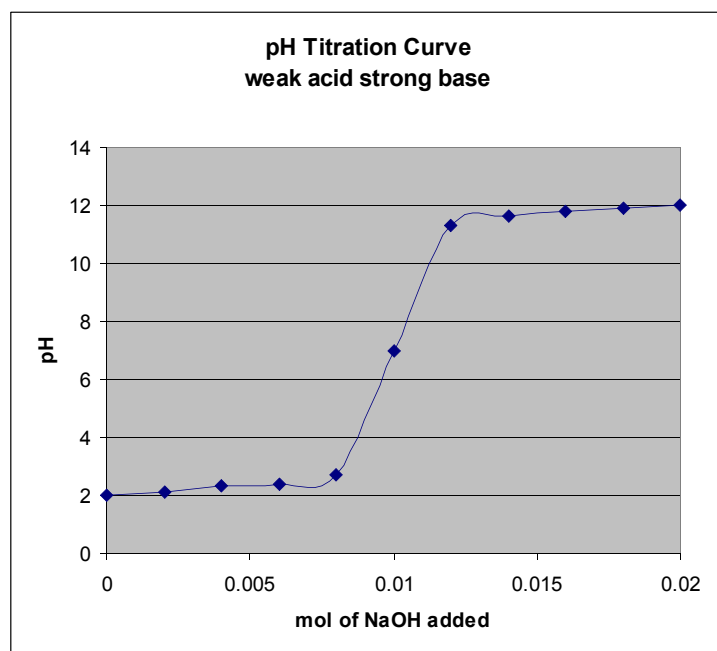
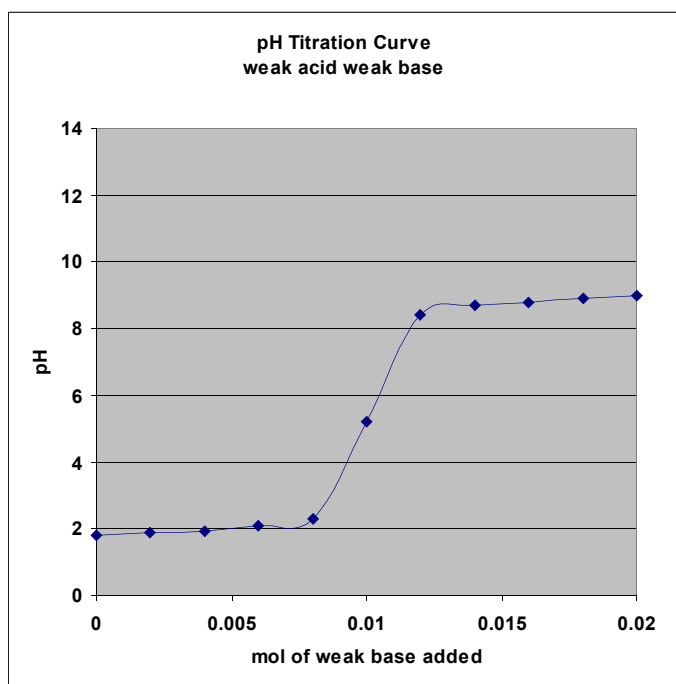
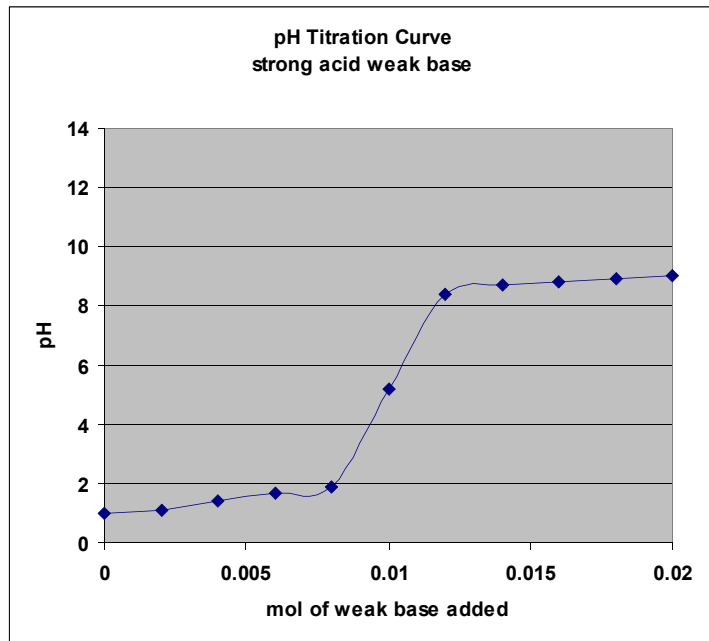
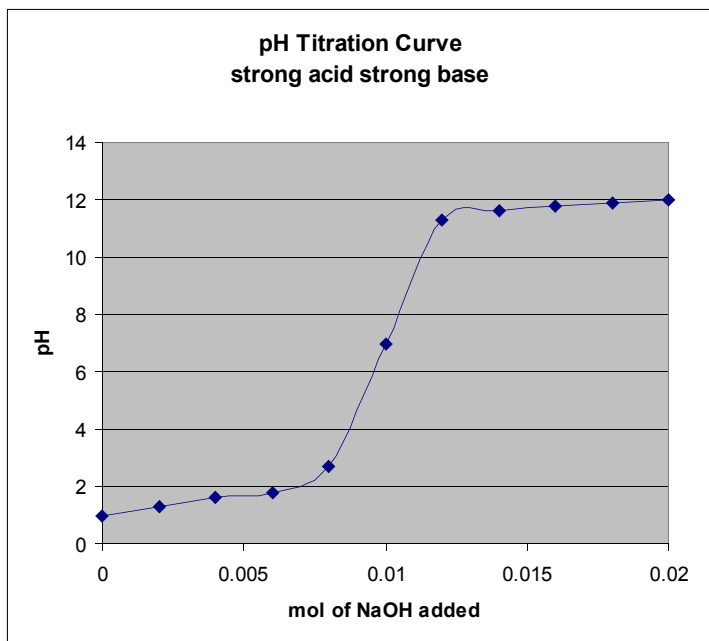


av titre	pH change/vol change
23.5	0.4
23.7	0.35
23.9	0.5
24.1	0.5
24.3	2.5
24.5	1.5
24.7	1
24.9	0.5
25.1	0.5

Eg,

	Vol (cm³)		pH
	23.40		3.20
av. titre = 23.50	↓ vol. change = 0.20		↓ pH change = 0.08
	23.60		3.28
av. titre = 23.70	↓ vol. change = 0.20		↓ pH change = 0.07
	23.80		3.35

4.



The main differences are in terms of the change in pH at the end point. The change in pH is greatest for a strong acid/strong base. The start and end pH values also vary depending on the type and molarity of acid and base used.

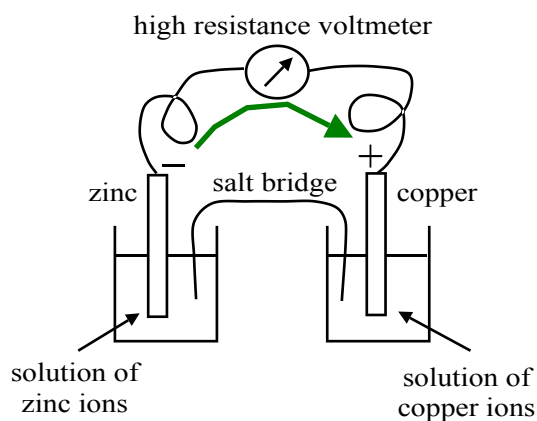
5. The type of acid given as unknown in this exercise is probably a weak acid. eg, ethanoic acid or maybe phosphoric acid. You should be able to tell from the shape of titration curve compared with those above.

Practical Seventeen:

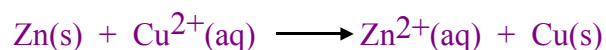
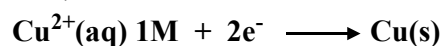
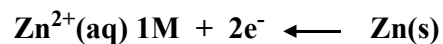
Redox reactions and Electrochemical cells.

Example of Experimental Results:

Battery 1.

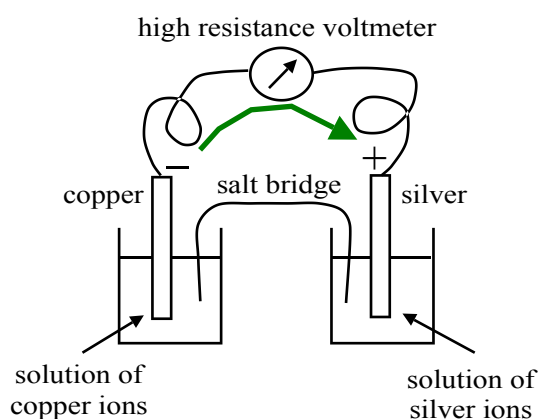


most negative electrode potential (-0.76v)

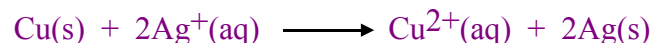
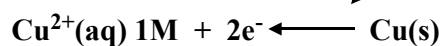


Experimental cell emf: 1.0v

Battery 2.

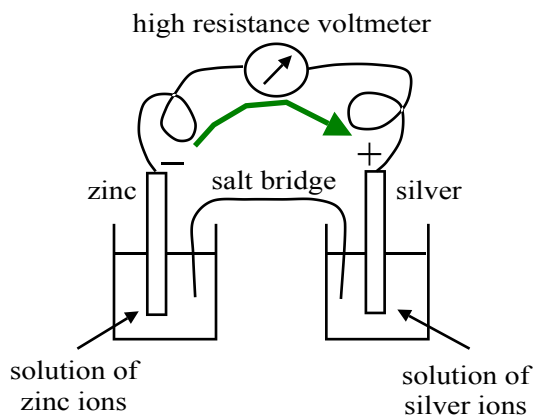


most negative (or least positive) electrode potential (0.34v)

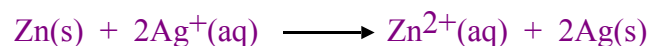
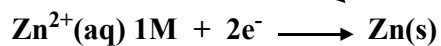


Experimental cell emf: 0.40v

Battery 3.



most negative electrode potential (-0.76v)



Experimental cell emf: 1.50v

Battery 1:

Experimental cell emf : 1.0v.

Theoretical cell emf: $0.34 - (-0.76) = 1.10\text{v}$

Battery 2:

Experimental cell emf: 0.4v

Theoretical cell emf: $0.80 - (0.34) = 0.46\text{v}$

Battery 3:

Experimental cell emf: 1.5v

Theoretical cell emf: $0.80 - (-0.76) = 1.56\text{v}$

The experimental results are in the right ball park but are low compared with the theoretical values. This is because the experimental conditions did not conform to strictly *standard* conditions. For example, the solutions may not have been exactly 1 molar or the temperature may not have been exactly 25°C or maybe the electrodes were not sufficiently clean or the voltmeter took current or the salt bridge was non ideal, etc.

Practical Eighteen:

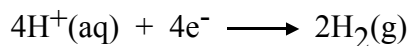
Electrolysis:

Example of Experimental Results:

Hoffmann Voltmeter:

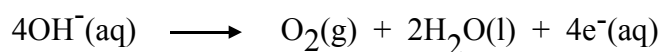
At the *negative* Pt electrode: a colourless gas collects which pops when ignited. This is hydrogen.

Reaction at the electrode: $\text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{H}^+(\text{aq}) + \text{OH}^-(\text{aq})$



At the *positive* Pt electrode: a colourless gas is evolved which re-lights a glowing splint. This gas is oxygen.

Reaction at the electrode: $\text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{H}^+(\text{aq}) + \text{OH}^-(\text{aq})$



Electrolysis of copper(II) sulphate.

At the anode (+): $\text{Cu}(\text{s}) \longrightarrow \text{Cu}^{2+}(\text{aq}) + 2\text{e}^-$

At the cathode (-): $\text{Cu}^{2+}(\text{aq}) + 2\text{e}^- \longrightarrow \text{Cu}(\text{s})$

1st experiment: Mass of clean fresh cathode: 10.20 g
Mass of clean fresh anode: 11.03 g
Mass of cathode after 25 mins at 1 amp = 10.69 g
Mass of anode after 25 mins at 1 amp = 10.54 g
Mass gain by cathode = 0.49 g
Mass loss by anode = 0.49 g

Within experimental error the gain and loss are the same (as expected).

2nd experiment: Mass of clean fresh cathode: 10.18 g
Mass of clean fresh anode: 11.02 g
Mass of cathode after 25 mins at 1.5 amp = 10.91 g
Mass of anode after 25 mins at 1.5 amp = 10.28 g
Mass gain by cathode = 0.73 g
Mass loss by anode = 0.74 g

From the above equations we see that to deposit 1 mole of copper (ie, 63.5 g) on the cathode requires 2 mole of electrons. This would require the passage of $2 \times 96\,480$ coulomb of electric current.

In the first experiment, $25 \times 60 \times 1$ coulomb (1500 C) was passed. We therefore expect, $(1500 / 2 \times 96\,480) \times 63.5$ g of copper to be deposited on the cathode (ie, 0.494 g). We expect the same amount to be removed from the anode. The experimental results confirm this.

In the second experiment $25 \times 60 \times 1.5$ coulomb (2250 C) was passed. We expect 0.74 g copper deposited. This is close to the experimental result above.

Practical Nineteen: Qualitative Organic Analysis: Example of Experimental Results:

The unknown pure substance (code: 83292) was a white crystalline solid.

Test	Observation	Inference
solubility	The substance was sparingly soluble in cold water but soluble in IMS.	Possible low molecular mass molecules with some polar character.
Melting point	The crystalline material melted sharply at 128°C.	(Subsequent tests indicate benzenecarboxamide (benzamide). This melting point supports that conclusion.
Sodium fusion test	<p>Tests for halogens proved negative.</p> <p>The test for nitrogen was positive (a blue colouration and precipitate of <i>prussian blue</i> was formed).</p> <p>Prussian blue: KFe(III)[Fe(CN)₆] (ie, iron(III) potassium hexacyanoferrate(II))</p>	<p>This clearly eliminates a whole raft of compounds both those in which the halogen is ionically bonded and those in which it is covalently bonded.</p> <p>This result suggests that the unknown is an amine or amide (simple or substituted).</p> <p>The result also confirms the presence of carbon because, in this test, the nitrogen is detected as cyanide (CN).</p>
Combustion	<p>A few crystals of the unknown were burnt on a crucible lid under a fume hood.</p> <p>The substance burnt with a very smokey flame and left no residue.</p>	<p>An <i>aromatic</i> substance is indicated.</p> <p>No residue indicates that the substance does not contain a metallic element.</p>
10% sodium hydroxide solution.	No apparent reaction in the cold, however, on warming the unknown dissolved and a pungent smelling gas was observed. The gas was alkaline to red litmus and gave white fumes with the vapour from the concentrated HCl bottle	A simple amide is indicated. The gas was ammonia.
Heat at 120°C for 15 mins in 75% sulphuric acid.	<p>On cooling the reaction mixture in iced water a white crystalline solid was formed. This was filtered off, recrystallised from IMS, dried and its melting point measured.</p> <p>The solid melted at 121°C.</p>	<p>The simple amide is undergoing hydrolysis to the corresponding carboxylic acid and an ammonium salt. The melting point of the carboxylic acid indicates it to be benzenecarboxylic acid (benzoic acid).</p> <p>The amide must be benzene-carboxamide (benzamide, C₆H₅CONH₂)</p>
Infra red spectroscopy confirmation. (KBr disc)	The ir of the unknown is identical to that of pure benzamide. Major peaks occurring at, 3380, 3180, 1660, 1630 & 1400 cm ⁻¹	<p>3380 & 3180 N-H str</p> <p>1660 C=O str</p> <p>1630 N-H bend</p> <p>1400 N-H bend.</p>

Practical Twenty.

Estimating the molarity of potassium chloride solution by back titration.

Example of Experimental Results:

The general procedure is to treat a known volume of the chloride solution with a known excess of silver nitrate solution. This converts all the chloride ion to insoluble silver chloride. This mixture is then filtered and a known volume of the filtrate titrated against potassium thiocyanate solution using iron(III) ammonium sulphate solution as indicator. This indicator gives a blood red colour when all of the silver has been precipitated as insoluble silver thiocyanate.

Procedure:

50.0 cm³ of the potassium chloride solution was pipetted into a 250 cm³ volumetric flask. 150.0 cm³ of 0.10 M silver nitrate was added. The mixture was acidified with 3 cm³ of concentrated nitric acid and made up to volume with distilled water and then shaken. The flask was allowed to stand so that the precipitate settled and then the mixture was filtered and the filtrate retained.

25.0 cm³ aliquots of the filtrate were titrated against 0.10 M potassium thiocyanate using iron(III) indicator.

Start volume cm ³	Finish volume cm ³	Volume delivered cm ³
0.00	14.55	14.55
0.00	14.70	14.70
0.00	14.60	14.60
Mean titre: 14.62 cm ³		

$$\text{mol of silver used} = \frac{150 \times 0.10}{1000} = 0.015 \text{ mol}$$

$$\text{mol of CNS}^- \text{ used in the titration} = \frac{14.62 \times 0.10}{1000} = 0.001462 \text{ mol}$$

$$\begin{aligned} \text{mol of CNS}^- \text{ required for } 250 \text{ cm}^3 &= 10 \times 0.001462 = 0.01462 \text{ mol} \\ &= \text{mol of silver ion left over (in excess)} \end{aligned}$$

$$\text{mol of silver ion reacted with chloride ion} = 0.015 - 0.01462 = 3.8 \times 10^{-4} \text{ mol}$$

this quantity of chloride ion is contained in 50 cm³ of potassium chloride solution
therefore molarity = (1000/50) × 3.8 × 10⁻⁴ mol dm⁻³

$$\begin{aligned} &= 7.6 \times 10^{-3} \text{ mol dm}^{-3} \\ (\text{g dm}^{-3} &= 74.6 \times 7.6 \times 10^{-3} = 0.522 \text{ g}) \end{aligned}$$

Practical Twenty One

Investigating Transition Metals

Example of Experimental Results:

V, Cr, Fe, Cu

metal	appearance	Melting Point °C	Density g cm ⁻³	ore	application
Vanadium	silvery	1915	6.11	In lead ores as Pb ₃ (VO ₄) ₂	ferrovanadium alloy and various catalysts
Chromium	shiny, silvery	1900	7.14	chromite FeCr ₂ O ₄	ferrochrome alloy and electroplating
Iron	grey, shiny	1535	7.87	haematite Fe ₃ O ₄	steel making
Copper	salmon (pink-orange) colour	1083	8.95	chalcopyrites CuFeS ₂	electrical conductor (wire & terminals)

test	observation	interpretation		
Vanadium				oxidation state
dioxovanadium(V) plus Zn (reducing agent)		Colour change	VO ₂ ⁺ (aq) ion	+5
			VO ²⁺ (aq) ion	+4
			V ³⁺ (aq) ion	+3
			V ²⁺ (aq) ion	+2
Chromium				
(a) (NH ₄) ₂ Cr ₂ O ₇	decomposes exothermically throwing up a greenish grey powder. Looks like a mini volcano!	(NH ₄) ₂ Cr ₂ O ₇ → Cr ₂ O ₃ (s) + N ₂ (g) + 4H ₂ O(g) greenish solid		
(b) Cr ³⁺ (aq) + violet colour NaOH(aq)	green gelatinous precipitate. Soluble in excess.	Cr ³⁺ (aq) + 3OH ⁻ (aq) → Cr(OH) ₃ (s) * Cr(OH) ₃ (s) + 3OH ⁻ (aq) → [Cr(OH) ₆] ³⁻ dark green solution *(Note: although often represented as the hydroxide the green precipitate is hydrated Cr ₂ O ₃ oxide)		

Chromium chemistry continued:

test	observation	interpretation
(b) continued: With ammonia solution	gelatinous green precipitate insoluble in excess.	green precipitate is often represented as the hydroxide but it is hydrated Cr(III) oxide.
(c) $\text{CrO}_4^{2-}(\text{aq})$ (yellow) barium chloride solution	yellow precipitate soluble in $\text{HCl}(\text{aq})$	barium chromate (BaCrO_4)
repeat with silver nitrate solution.	brick red precipitate soluble in nitric acid	silver chromate (Ag_2CrO_4)
(d) $\text{Cr}_2\text{O}_7^{2-}(\text{aq}) + \text{H}^+(\text{aq})$ plus $\text{Fe}^{2+}(\text{aq})$	Initially the iron(II) sulphate solution is pale green - its solution gradually turns yellow	Potassium dichromate in acid solution is a powerful oxidising agent. It oxidises green iron(II) to the yellow iron(III) ion,
$\text{Cr}_2\text{O}_7^{2-}(\text{aq}) + \text{H}^+(\text{aq})$ + $\text{KI}(\text{aq})$	Colourless solution turns brown	Iodide is oxidised to iodine (brown in aq solution)
$\text{Cr}_2\text{O}_7^{2-}(\text{aq})$ + $\text{OH}^-(\text{aq})$ then add acid	As alkali is added the dichromate solution turns yellow. Addition of acid restores the orange colour characteristic of dichromate.	A solution of dichromate is an equilibrium mixture: $\text{Cr}_2\text{O}_7^{2-}(\text{aq}) + \text{H}_2\text{O}(\text{l}) \rightleftharpoons 2\text{CrO}_4^{2-}(\text{aq}) + 2\text{H}^+(\text{aq})$ <div style="display: flex; justify-content: space-around; width: 100%;"> orange yellow </div> When alkali is added the equilibrium moves to the right and yellow chromate increases. Adding acid reverses the process converting chromate back into dichromate.
Iron		
(a) A solution of iron(II) sulphate.	Freshly made this is pale green. On standing the surface of the solution turns yellow-brown.	Iron(II) is being oxidised by the air to iron(III). $\text{Fe}(\text{OH})_2$ (readily oxidised to brown $\text{Fe}(\text{OH})_3$) $\text{Fe}(\text{OH})_2$ Fe^{2+} are being reduced to iron so removing the characteristic green colour due to hydrated Fe^{2+} ions.
With $\text{NaOH}(\text{aq})$	Light green precipitate. Insoluble in excess.	
With dil. ammonia solution.	Light green precipitate. Insoluble in excess.	
With Zn	The light green solution gets gradually lighter in colour.	
(b) Acid + permanganate	The purple colour of the permanganate is dispersed and the iron(II) solution becomes more yellow.	Permanganate is a powerful oxidising agent and rapidly converts iron(II) (green) into iron(III) (yellow-brown). The permanganate is converted to colourless Mn^{2+} ions.

Iron chemistry continued:

test	observation	interpretation
<p>(c)</p> <p>Test iron(III) solution for acidity</p> <p>Iron(III) solution + NaOH(aq)</p> <p>Iron(III) solution + NH₃(aq)</p>	<p>The solution is acid (about pH 5)</p> <p>yellow-brown (rust coloured) gelatinous precipitate formed. Insoluble in excess.</p> <p>Yellow-brown precipitate formed. Insoluble in excess.</p>	<p>The hydrated iron(III) ion undergoes deprotonation providing an equilibrium mixture containing hydrogen ion.</p> <p>Fe(OH)₃</p> <p>Fe(OH)₃</p>
<p>(d)</p> <p>Add dil. potassium ferricyanide (hexacyanoferrate(III)) solution to iron(II) solution.</p>	<p>Dark blue precipitate formed.</p>	<p>This is Turnbells blue (also called prussian blue)</p> <p>KFe(II)[Fe(III)(CN)₆]</p>
<p>(e)</p> <p>Add dil. potassium thiocyanate solution to a solution of iron(III).</p>	<p>Blood red colouration.</p>	<p>The blood red colour is due to a mixture of complex ions:</p> <p>[Fe(SCN)(H₂O)₅]²⁺, Fe(SCN)₃ & [Fe(SCN)₄]⁻</p>
Copper		
<p>(a)</p> <p>Colour of copper(II) sulphate solution?</p> <p>With dil. sodium hydroxide.</p> <p>With dil. ammonia solution.</p>	<p>Pale blue solution.</p> <p>Pale blue precipitate. Insoluble in excess.</p> <p>Pale blue precipitate. Soluble in excess to give a dark blue solution.</p>	<p>Due to hydrated Cu²⁺ ions.</p> <p>Cu(OH)₂</p> <p>Cu(OH)₂</p> <p>The dark blue colour is due to the complex ion:</p> <p>[Cu(NH₃)₄(H₂O)₂]²⁺</p>
<p>(b)</p> <p>Add KI(aq) to copper sulphate solution.</p>	<p>A precipitate and dark brown solution is formed.</p> <p>Isolation of the precipitate by filtration shows that it is white.</p>	<p>The brown colour is due to iodine and the white precipitate is copper(I) iodide.</p> <p>2Cu²⁺(aq) + 4I⁻(aq) = Cu₂I₂(s) + I₂(aq)</p>
<p>(c)</p> <p>Add zinc to a dil. solution of copper(II) sulphate.</p>	<p>The blue colour of hydrated copper(II) ion gradually disappears.</p>	<p>This happens because the zinc reduces the blue, hydrated, copper(II) ion to copper (which is then deposited)</p>
<p>(d)</p> <p>Copper + conc. HNO₃</p>	<p>A vigorous reaction takes place. A blue solution is formed and a brown gas is evolved.</p> <p>The gases from the reaction mixture relight a <u>glowing</u> splint.</p>	<p>The acid oxidises the metal to copper(II). Nitrogen(IV) oxide is produced. This gas will support the combustion of a glowing splint providing the splint is hot enough.</p> <p>Cu(s) + 4H⁺(aq) + 2NO₃⁻(aq) = Cu²⁺(aq) + 2NO₂(g) + 2H₂O(l)</p>

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